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(19)

(54) COATING COMPOSITIONS

(71) We, RECKITT & COLMAN PRODUCTS LIMITED, a British Company, of P.O. Box 26, 1/17 Burlington Lane, London W4 RW, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to coating compositions and methods of making them, more particularly to dehydrated compositions containing emulsion polymers that are reconstitutable to aqueous dispersions.

In the fields of decorative and protective coating, extensive use is made of aqueous polymer latices, produced by emulsion polymerisation, as major film-forming ingredients in both permanent and temporary organo-coatings including certain floor coatings.

Polymers and copolymers produced by emulsion polymerisation are known as "emulsion polymers".

Typical aqueous coating compositions contain many additional ingredients. Thus in the formulation of paints pigments, extenders, suspending agents, stabilisers, levelling agents, plasticisers and emulsifiers may be used; in cleaning compositions detergents including detergent salts and builders are used; and in emulsion polish compositions, such as are used for floor care there may be included, for example, alkali-soluble resins, surfactants, fugitive and non-fugitive plasticisers as well as waxes.

Frequently, emulsion polymer latex is manufactured at one site then transported to another where it is subsequently formulated into a coating composition. Quality control problems arise, often through environmental changes during the transportation of the latex. Moreover, transportation costs are high and since emulsion polymer latices are frequently produced at a solids content of up to 50%, frequently 10—25%, the nett result is the expensive transportation of water. It has now been found that certain latices may be dehydrated for subsequent rehydration and recovery of useful dispersions.

According to the present invention there is provided a particulate dehydrated composition reconstituted to a dispersion on mixing with aqueous base and comprising emulsion polymer, water-dispersible wax and emulsifier.

Preferably the emulsion polymer is metallised with a polyvalent metal ion, such as an ion derived from magnesium, calcium, strontium, chromium, manganese, iron, cobalt, nickel, copper, zinc, zirconium, cadmium or tin.

Preferably the composition additionally contains an alkali-soluble resin.

Preferably the composition additionally includes a solid coalescent aid such as caprolactam.

In a preferred embodiment a composition reconstitutable to a dispersion on mixing with water comprises emulsion polymer, water dispersible wax, an emulsifier and sufficient base to cause dispersion.

The base is preferably a carbonate or bicarbonate salt of ammonium, sodium or potassium or a mixture thereof.

Alternatively, the base is an amine carbonate or carbamate or a mixture thereof.

The preferred amines are:- 2-amino-2-methylpropan-1-ol 3 methoxypropylamine, di-isopropylamine, ethylene diamine or morpholine.

The emulsion polymer preferably has an acid content of from 3.8% to 29.5% w/w based on the total monomer mix and expressed as acrylic acid.

Preferably the acid content is from 5.0% to 29.0% w/w based on the total monomer mix and expressed as acrylic acid.

Preferably the emulsion polymer has a minimum film-forming temperature (hereinafter referr d to as the "MFT") greater than 15°C, conveniently in the rang 20°C to 80°C.

Preferably the emulsion polymer contains methacrylic acid as the principle acidic monomer; more especially methacrylic acid represents at least 6% of the monomeric mixture from which the emulsion polymer is prepared.

The emulsion polymer is preferably a copolymer of styrene with a mixture of lower α β-unsaturated carboxylic acids.

The water-dispersible wax is preferably a polyethylene wax and preferably the composition contains up to 20% w/w of said wax.

In one preferred embodiment the composition contains up to 80% w/w of emulsion polymer, up to 20% w/w water-dispersible wax, 0—8% w/w plasticiser, up to 0.05% w/w levelling aid and the composition has MFT in the range 15°C to 28°C.

The invention also envisages a method for preparing a solid dehydrated coating composition comprising the step of deyhdrating, at a temperature less than the minimum film-forming temperature characteristic of the composition, an aqueous dispersion containing emulsion polymer, water-dispersible wax and emulsifier.

Preferably, the dehydration step is carried out whilst the temperature is above ambient temperature but below the MFT of the composition.

The dispersion is preferably dehydrated by air-drying on shallow trays or by spray-drying.

Optionally a particulate solid material such as a detergent salt, for example the alkaline materials:- ammonium, sodium or potassium carbonate, bicarbonate, phosphate or silicate is included in the dehydrated composition.

One method of making a dehydrated composition comprises the steps of spray drying the dispersion composition whilst simultaneously coating the particulate solid material therewith. Conveniently, this is effected by supporting the particulate solid material on a fluidised bed whilst the composition is spray coated thereon.

If during dehydration the temperature of the dispersion or dehydrate is allowed to rise above the characteristic MFT of the composition, a sticky product can then result presenting handling problems and difficulties may be experienced in redispersing the dehydrate. After total dehydration of a dispersion en masse a more or less hard cake results which may be broken up and powdered for redispersion. Whilst the preferred embodiment proposes spray-drying any suitable means of drying (such as air-drying, freeze-drying, oven-drying) may be employed.

As described above the invention envisages spray-drying a dispersion whilst simultaneously coating it onto an existing particular solid material contained in a fluidised bed within the spray-drying apparatus. This method may be conveniently employed when for example, a particulate cleaning composition is to be prepared comprising one or more detergent salts and suitable polymeric materials. The intention with such compositions is to disperse the dry particles in water and apply the so-formed dispersion to a surface to be cleaned with an applicator. Cleansing occurs through the action of the detergent salts component, soil being removed in the applicator. Upon drying, a shine due to the residual polymeric material coating the surface, remains after final air-drying.

Valuable dehydrates may be obtained with emulsion copolymers of lower α β-ethylenically unsaturated monocarboxylic acids such as acrylic, methacrylic, crotonic and such acids as itaconic acid which are usually used in admixture and may be modified by incorporating other monomers or by blending with other polymers. The polymer latices are produced via emulsion polymerisation which may be conducted in a single step using a homogeneous blend of monomers, or in a polyphase polymerisation in which the polymerisation of a group of monomers is interrupted and a further group of monomers is added and polymerised and so on, resulting in either a "blend" of polymeric types or a "core" polymer depending on the precise conditions of reaction as is well known. It is not essential that the carboxyl group be located externally of the individual polymer particles, for example in the case of "core" polymers.

Preferred emulsion polymers have a relatively high carboxyl content, that is free —COOH groups, or such groups neutralised by salt formation. Preferably the carboxylated copolymers contain at least 6% w/w of acid based on monomer mix.

So far it has not been practicable to produce an emulsion copolymer that contains more than about 35% w/w acid.

It has been found that low acid-content copolymers lead to dehydrates that redisperse unsatisfactorily. At an acid-content level of about 3% some redispersion occurs but insufficient for convenience.

Broadly speaking the acid value of emulsion polymers and copolymers suitable for use in the present invention will lie in the range 30 to 230 preferably 39 to 228 which ranges respectively approximately correspond with an acid content, expressed as acrylic acid, of 3.8 to 29.5% w/w and 5.0 to 29.2% w/w.

Other monomers that may be copolymerised with the α β-ethylenically unsaturated monocarboxylic acids are typically:- styrene; substituted styrenes such as α-methyl styrene and halogenated styrenes; lower alkyl methacrylate esters and/or acrylonitrile which modify the final copolymer by increasing its hardness: higher alkyl acrylates from alcohols containing up to 12 carbon atoms in straight or branched chains and cyclic alcohols; alkyl methacrylates from alcohols containing up to 12 carbon atoms notably butyl- and 3-ethylhexyl methacrylate which act by way of internal plasticisers modifying the hardness and film-forming properties of the final copolymer: hydroxylates; acrylamides for example methylol acrylamide; epoxidated monomers such as glycidyl acrylate and -methacrylate; aminoalcohol acrylate such as dimethylaminoethanol acrylate; divinyl benzene. These monomers may be added singly or in combination at levels often less than 0.1 w/w of total monomer mix up to about 15% w/w to impart special properties to the final copolymer, for example to improve intercoat adhesion, adhesion to substrates: self gloss, durability, detergent resistance and rubber heel marking.

Preferably, the emulsion polymer is "metallised", that is the latex includes a polyvalent metal salt in solution which is believed to cause "reversible" cross-linking between polymer molecules upon drying out of the latex. By "reversible" is meant that under certain conditions cross-linking occurs, for example upon a film drying out; but by changing the pH conditions, for example by addition of an aqueous base, such as ammonia or aliphatic amines or their salts, to the applied dried coating the cross-linkages may be broken. Only in the cross-linked condition is the polymer insensitive to water and detergent. Such metallised latices are well known especially in the field of floor maintenance where the latices are utilised in floor coating compositions for their water- and detergent-resistance coupled with ease of removal when desired.

An important factor influencing MFT and solubility, hence redispersibility, of polymeric materials, apart from the selection of the precise chemical profile for monomers in a copolymer, is molecular weight. To some extent molecular weight may be controlled during polymerisation by the use of chain transfer agents, such as the halogenated hydrocarbons like carbon tetrachloride and carbon tetrabromide, and mercaptans like 'tert'-dodecyl mercaptan. Mean molecular weight values may be estimated from viscosity characteristics measured with the Ostwald viscometer using standardised solution of polymeric material in organic solvents for comparison.

The present invention has been found to be applicable to the dehydration of latices containing both high and low molecular weight polymers and copolymers. Redispersion is favoured with polymers of relatively low molecular weight. Those versed in the art of formulating floor polished will appreciate that an alkali-soluble resin component, needed to optimise gloss and levelling, may be exemplified by a copolymer prepared from some of the abovementioned monomers. In which case the molecular weight is necessarily low to achieve the desired solubility in alkali.

In greater detail emulsion floor polish compositions, which may be pigmented or unpigmented, have solids content of about 8% minimum and typically contain one or more of the following component types:

(i) metallised copolymer emulsion,

(ii) non-fugitive plasticiser,

(iii) coalescent solvents or fugitive plasticiser,

(iv) surface active agents,

(v) water-dispersed wax,

(vi) dissolved alkali-soluble resin.

Coating compositions are prepared from particulate dehydrated compositions of the present invention by dispersing dehydrate in water containing a suitable base and optionally plasticiser ingredient.

To prepare a dehydrate which is capable of protracted storage for a polish formulation it is important that the coalescent solvent, examples of which are diethyleneglycol monomethylether or monoethyl ether and fugitive plasticisers be omitted. Still retaining these two component types, a product can be achieved with adequate coating properties for some purposes upon reconstitution, for example, by use of a powder form of coalescent solvent or coalescent aid such as caprolactam; difficulty may be experienced in reconstituting because of the sticky nature occurring or developing in such dehydrates after standing some time.

Reconstituted emulsion polish compositions completely lacking coalescent solvent may be employed for renovating purposes upon a previously coated floor. In such application the formulation needs to be such that the MFT of the composition is exceeded during a final buffing step which usually attains a minimum of 28°C on the floor, after application and during drying in situ. It is stressed that in these circumstances no coalescent solvent is necessary.

Such a composition has the advantage that if sprayed onto furniture or drapes and/or carpet, the composition dries to a powder which may be simply dusted off or brushed off as any other dusty material. In use the process, which is variously known as "spray-buffing", "spray-polishing" or "speed-polishing", involves applying, as by spraying, a wet coating onto the floor and before the coating has dried, buffing with a high-speed polishing machine to provide a finish which is a dry, hard, glossy surface often with a mirror-like appearance. The process obviates frequent stripping of the substrate and polishing brushes do not soil rapidly with a composition containing no coalescent solvent.

Essentially, the composition designed for such a purpose contain insufficient plasticiser and/or coalescent solvent to render the composition, as a whole, film-forming at the relatively low temperatures experienced at floor level. Whilst there is a requirement for a minimum concentration of plasticiser, the coalescent solvent component need not be present in such compositions.

A particularly useful range of bases are the so-called carbonates or carbamates of organic amines. These are the material forming a crust that sometimes surrounds the stopper of bottles containing amines in storage places.

Such materials may be prepared by interacting free amine and carbon dioxide for example in solid form.

The amine carbonates or carbamates being solids may be admixed as such into dehydrated polish composition for transportation so that reconstitution does not necessitate separate addition of base.

The rate of dispersion depends upon the temperature of the mixing, particle size of the dehydrate and the desired concentration, that is solids content, of the final dispersion.

The following are some non-limitative examples which save for Examples 1, 2 and 3 further illustrate the invention; Examples 1, 2 and 3 serve to illustrate the method of carrying out some aspects of the invention and the preparation of some starting materials.

Example 1.

36% solids content copolymer latices A, B, C and D were prepared in conventional apparatus by emulsion polymerisation at atmospheric pressure. 2.8% w/w dodecylated oxydibenzene disulphonate sodium salt was used as anionic surfactant and 1% w/w ammonium persulphate was used as free radical initiator. The compositions of A, B, C and D are shown in Table 1.

TABLE 1

Polymer	Compositions of monomer mix Parts w/w per 100 parts mixture					Percentage w/w on total monomer TDM	MFT °C of metallised polymer
	MAA	MMA	BA	STY	GM		
A	12	39	29	20	–	0.75	59
B	12	59	29	–	–	1.75	50
C	12	58	29	–	1	1.75	49
D	12	48	40	–	–	1.50	43

Legend: MAA = Methacrylic acid

MMA = Methyl methacrylate

BA = Butyl acrylate

STY = Styrene

GM = Glycidyl methacrylate

TDM = 'tert' -dodecyl mercaptan, a chain transfer agent for control of molecular weight

The dispersions of emulsion polymers A, B, C and D were obtained as acidic bluish white, mobile fluids, containing less than 0.2 w/w unreacted monomer.

Each of the emulsion polymers was "metallised" by mixing with 25.170 parts zinc ammonium acetate solution per 100 parts dispersion. The zinc ammonium acetate solution was prepared by mixing 2.101 parts zinc acetate dihydrate, 2.166 parts aqueous ammonium hydroxide (S.G. 0.880), 0.614 parts nonyl phenyl polyoxyethanol in 15.299 parts water. This provides for roughly 50% of the free carboxyl groups to be "metallised" or cross-linked.

Finally the dispersions were adjusted to 30% solids content and pH 7.8—8.8. No separation of phases was observed on centrifuging at 4000 r.p.m. for 10 minutes.

The emulsion polymer dispersions so produced were air-dried on shallow trays at a temperature of below the MFT of the metallised emulsion polymer. Virtually odourles, hard cakes were produced and on crushing to pass a 100 mesh (BS 410) sieve became white powders.

The dried products when stirred with water alone do not disperse or emulsify, illustrated by the fact that two phases are readily separable. Raising the pH of the mixtures to above 7.8 by addition of ammonium hydroxide solution results in bluish white dispersions exhibiting no separation on centrifuging at 4000 r.p.m. for 10 minutes.

The product dried to a clear, shiny coating when spread over a test surface.

Example 2.

The emulsion polymer dispersions prepared in Example 1 were intoduced into a NIRO ATOMISER spray drying unit operating under the following conditions.

Air (inlet) temperature 200°C

Air (outlet) temperature 70°C

Atomiser speed 60,000 r.p.m.

By this method extremely fine particle size powders were produced estimated at between 1 and 8 microns mean particle size.

The products when stirred with water alone do not disperse or emulsify, illustrated by the fact that the two phases are readily separable. Raising the pH of the mixtures to 7.8 by addition of ammonium hydroxide solution results in bluish white dispersions exhibiting no separation on centrifuging at 4000 r.p.m. for 10 minutes.

The product dried to a clear, shiny coating when spread over a test surface.

Example 3.

A coating composition was prepared using the dried emulsin polymer A from Example 1. 78.79 parts water, 4 parts diethylene glycol monomethyl ether and 0.5 parts of 0.880 ammonium hydroxide were stirred while adding 13.13 parts of the powder over 20 minutes. After stirring for a further 10 minutes period 0.79 parts dibutyl phthalate and 0.79 parts tributoxyethyl phosphate were added. Stirring was continued until complete redispersion of the ingredients was obtained. This could be detected by examination of the dispersion on a black Test card for signs of bittiness and also by centrifuging a small portion at 4000 r.p.m. for 10 minutes. After stirring the dispersion for a total of one hour no signs of bittiness or separation after centrifuging was noted.

The dispersion when coated onto a test card dried to a glossy, water resistant, flexible polymeric coating.

Example 4.

A dispersion of an intermediate molecular weight oxidised polyethylene wax was prepared by the following procedure:

Using a pressure emulsification vessel at 150°C a mixture of 19.455 parts polyethylene wax AC 392, 4.864 parts Lissapol NXP (Registered Trade Mark), 0.468 parts potassium hydroxide, 0.195 parts sodium metabisulphite and 46.429 parts softened water was stirred for one hour. The emulsion was cooled to give a stable dispersion of total solids content of 35%.

The dispersion was mixed with metallised polymer B shown in Table 1 in the following ratio:

Polymer dispersion	139.9
Wax dispersion	22.5

The polyer/wax dispersion had a solids content of 30.69%, pH 7.8 and MFT 44°C.

The dispersion was spray dried using the conditions described in Example 2 to give a fine, white powder.

Using the method described in Example 3 the powder was redispersed to give a fine particle size, stable emulsion.

A 2.0 thou wet thickness film was cast on to a glass panel to give a clear, tough, water-resistant film on drying. The presence of the wax component improved the scratch resistance of the coating film.

Example 5.

A dispersion of low molecular weight oxidised polyethylene wax was prepared by the following procedure:

Using a pressure emulsification vessel at 140°C a mixture of 19.455 parts polyethylene wax AC 540, 4.864 parts Lissapol NXP, 0.486 parts potassium hydroxide, 0.195 parts sodium metabisulphite and 46.429 parts softened water was stirred for one hour. The emulsion was cooled to give a stable dispersion of total solids content of 35%.

The dispersion was formulated into a coating composition using the following recipe:—

Metallised Polymer dispersion (B, Table 1)	188.5	parts
AC 540 wax dispersion	30.3	parts
Tributoxyethyl Phosphate	4.6	parts
Fluocarbon Surfactant	0.035	parts

The dispersion was then spray dried on a NIRO Atomiser unit fitted with an Air Broom assembly. The inlet temperature was 185°C and the outlet temperature 70°C. A fine white powder was collected.

Separate portions of the powder were mixed with th following alkaline substances

(i) Ammonium hydroxide (SG 0.880)

(ii) Ethylene diamine

(iii) Ammonium carbonate

and dispersed by stirring into water to give a final total solids content of 10% by weight and a final pH of 8.3—9.2. The dispersions showed no separation after centrifuging for 10 minutes at 4000 r.p.m.

The emulsions were used for the routine maintenance of previously trafficked floor substrates such as vinyl, lino, vinyl asbestos and sealed wood by the process known as "Spray Cleaning". In this process a film of the emulsion is sprayed on the floor and then immediately burnished to a shine using a floor polishing machine.

Example 6.

Two alkali-soluble resin solutions were prepared as follows:

(a) Heat and stir at 55°C a mixture of 2.35 parts ammonium hydroxide solution (SG 0.88) and softened water (80.3 parts) in a vessel. Add 15 parts modified styrene maleic anhydride resin (SMA 17352A) over 15 minutes and then a further 2.35 parts ammonium hydroxide solution. A clear solution of 15% solids is obtained after cooling.

(b) Heat and stir at 80°C a mixture of 2.75 parts ammonium hydroxide solution (SG 0.88) and 72.5 parts softened water in a vessel. Add 22 parts of a rosin based resin SR88, a Trade Mark of Schencetady, over 15 minutes and then a further 2.75 parts ammonium hydroxide. A clear solution of 22% solids is obtained after cooling.

Using these resin solutions a series of dispersions utilising the wax and metallised polymer dispersions previously described were prepared by mixing. The compositions are shown in Table 2.

Two series of powders were prepared by (1) drying the dispersions on trays below their MFT's and (2) by spray drying the dispersions in a NIRO spray drying unit.

The theoretical composition of the powders excluding the excess of zinc salt and emulsifiers present in the metallised polymer and wax dispersions is:

zinc metallised powder	82.686%
Polyethylene wax	12.937%
alkali-soluble resin	4.311%
fluorocarbon surfactant	0.066%

The powders were redispersed in alkaline medium to a level of 15% total solids using the following formulation:

Powder	14	parts by weight
Dipropylene glycol monomethyl ether	2.132	parts by weight
Diethylene glycol monomethyl ether	2.132	parts by weight
Dibutyl phthalate	0.132	parts by weight
Tributoxyethyl phosphate	0.843	parts by weight
Ethylene diamine	0.800	parts by weight
Softened water	83.812	parts by weight
TOTAL	104.560	

The method of dispersion comprised stirring at 50°C a mixture of the water, ethylene diamine, dipropylene glycol monomethyl ether, diethylene glycol monomethyl ether and then adding over 45 minutes the powder. After stirring for a further 30 minutes and then cooling a stable dispersion was obtained. This showed no separation on centrifuging.

The coatings were applied in the normal manner to flooring substrates such as vinyl and vinyl asbestos tiles and assessed for gloss, levelling, recoatability and scrub resistance. In comparing the above composition with commercially available floor polishes excellent results were obtained. Also as expected with metallised floor polishes, the films were readily removed by dilute alkaline detergents.

TABLE 2

Metallised Polymer Dispersion at 30% Solids	Parts by Weight				
	1	2	3	4	5
A	139.94	139.94	139.94	139.94	139.94
B					
C					
D					
Polyethylene Wax Dispersions at 35% Solids					
AC 392 (Example 4)	22.52	22.52	22.52	11.56	22.52
AC 540 (Example 5)				11.26	
Alkali Soluble Resin					
Styrene-Maleic Anhydride Type at 15% Solids	17.51		17.51	17.51	17.51
Rosin-based resin at 22% Solids		11.92			
Fluorocarbon Surfactant FC 128 1% Solids Aqueous Solution	4.00	4.00	4.00	4.00	4.00
Total	183.97	178.4	183.97	183.97	183.97
Total Solids	28.55%	29.44%	28.55%	28.77%	28.77%
MFT °C	49°C	49°C	44°C	47°C	35°C

Example 7.

A 36% solids metallised emulsion polymer was prepared by addition at room temperature of 3.846 parts zinc acetate dihydrate, 5.615 parts ammonium hydroxide (0.880) and 3.964 parts 20% Ethylan (Registered Trade Mark) HA in 13.77 parts water to 100 parts of a dispersion of a 44% solids core polymer produced by split phase emulsion polymerisation at 75°C of a monomer mix consisting of 22% methacrylic acid, 29% butyl acrylate and 49% methyl methacrylate using tert-dodecyl mercaptan as chain transfer agent.

A polymer emulsion concentrate of constitution 89.63% of the above dispersion, 6.59% polyethylene wax emulsion (25% solids), 1.72% levelling aid (FC128) and 2.06% tributoxyethyl phosphate plasticiser was air-dried as a thin film at ambient temperature and powdered in a ball mill.

The dehydrate produced was intimately mixed with ammonium carbonate in weight ratio 85:15. The product was redispersed in cold tap water to give a final dispersion of 16% solids content.

On coating out this product, a non-buffable, dry-bright polish, an excellent finish was obtained after drying. Although no levelling resin was included, gloss and levelling were the same as that obtained with a commercially available liquid dry-bright polish but had slightly longer drying time.

Example 8.

In place of the ammonium carbonate used in Examples 7, 2-amino-2-methylpropan-1-ol carbonate/carbamate was mixed with the dehydrate produced in Example 7 in the ratio 87 parts dehydrate to 13 parts base.

The mixture was dispersed in cold tap water to provide a 16% solids dispersion of uniform consistency. On coating out, a dry film with excellent gloss and levelling characteristics was produced. After traffic and re-application of a fresh coating self-stripping properties were exhibited. In some tests superior gloss and levelling, as compared with a self-stripping polish currently available on the market, were observed.

Example 9.

To 89g 2-amino-2-methylpropan-1-ol dissolved in 600g 1.1.1-trichloroethane was added 22g solid carbon dioxide with stirring. The temperature of the mixture rose to 45°C and a white precipitate formed which was separated by filtration, suspended in 300 ml 40°—60°C petroleum spirit, filtered and dried under vacuum at ambient temperature. The white, crystalline product obtained was the carbonate/carbamate salt.

Using the same technique analogous salts with morpholine, diisopropylamine and 3-methoxypropylamine were prepared.

Example 10.

A 36% solids metallised emulsion polymer was prepared by addition at room temperature of 2.463 parts zinc acetate dihydrate, 2.552 parts ammonium hydroxide (0.880) and 4.575 parts 20% Sapogenat T500 in 12.388 parts water to 78.072 parts of a dispersion of a 44% solids core polymer produced by split phase emulsion polymerisation at 75°C. Of a monomer mix consisting of 12% methacrylic acid, 29% butyl acrylate and 59% methyl methacrylate using 'tert' dodecyl mercaptan as chain transfer agent.

A polymer emulsion concentrate of constitution 71.99% of the above dispersion, 24.29% polyethylene wax emulsion (20% solids), 2.1% tributoxyethyl phosphate and 1.62% fluocarbon levelling aid, FC128 (1% active) was prepared by cold blending. The blend was spray dried in a NIRO ATOMISER SPRAY DRY unit, fitted with an air broom, at air inlet temperature of 180°C.

The dried product was dispersed in aqueous ammonia to give 10% solids dispersion of pH9.

A wet, sprayed-on coating on a clear test surface was burnished with a high speed polishing machine to produce a durable finish with high sheen. This shows that at reduced plasticiser level a useful finish can be prepared by this method of the invention. Dried-on spots of spray were simply dusted off hard surfaces and could be shaken out of drapes. The level of plasticiser may be still further reduced if the polymer used has a higher concentration of methacrylic acid in the monomer mix than the 12% used in this Example. High methacrylic acid content is believed to improve the self-levelling characteristics of the final polymer and hence compositions containing it. Thus, in the range 15%—25% methacrylic acid content, no plasticiser is necessary for spray polishing application; from 5—15% methacrylic acid content the necessary concentration diminishes with increased acid concentration.

The application of the present invention to the production of reconstitutable wax-containing emulsion floor polishes is of great advantage because the dehydrated product is not sensitive to moisture during shipment being reconstitutable only in alkaline medium. Since only coalescent solvent and the base components are omitted there is potential saving on special facilities at remote sites otherwise needed to incorporate some components, for example plant for production of wax-dispersion and the like.

Utilising solid bases such as ammonium carbonate or amine carbonates/carbamates it is possible to prepare dry powders by mixing dehydrated coating compositions lacking base with such solid bases for packaging in moisture-proof containers. With such dry compositions the user may simply mix with water, especially in the case of compositions for use in the "spray-buffing" process.

WHAT WE CLAIM IS:—

1. A particulate dehydrated composition reconstitutable to a dispersion on mixing with aqueous base and comprising emulsion polymer, water-dispersible wax and emulsifi r.

2. A composition as claimed in Claim 1 in which the emulsion olymer is metallised with a polyvalent metal ion.

3. A composition as claimed in Claim 2 in which the polyvalent metal ion is derived from magnesium, calcium, strontium, chromium, manganese, iron, cobalt, nickel, copper, zinc, zirconium, cadmium or tin.

4. A composition as claimed in any preceding claim which additionally contains an alkali-soluble resin.

5. A composition as claimed in any preceding claim which additionally includes a solid coalescent aid.

6. A composition as claimed in Claim 5 in which the coalescent aid is caprolactam.

7. A particulate dehydrated composition reconstitutable to a dispersion on mixing with water comprising emulsion polymer, water dispersible wax, an emulsifier and sufficient base to cause dispersion.

8. A composition as claimed in Claim 7 in which the base is a carbonate or bicarbonate salt of ammonium, sodium or potassium or a mixture thereof.

9. A composition as claimed in Claim 7 in which the base is an amine carbonate, carbamate or mixture thereof.

10. A composition as claimed in Claim 9 in which the amine is 2-amino-2-methylpropan-1-ol.

11. A composition as claimed in any preceding claim in which the emulsion polymer has an acid content of from 3.8% to 29.5% w/w based on the total monomer mix and expressed as acrylic acid.

12. A composition as claimed in any preceding claim in which the emulsion polymer has an acid content of from 5.0% to 29.0% based on the total monomer mix and expressed as acrylic acid.

13. A composition as claimed in any preceding claim in which the emulsion polymer has MFT greater than 15°C.

14. A composition as claimed in Claim 13 in which the emulsion polymer has MFT in the range of 20°C to 80°C.

15. A composition as claimed in any preceding claim and of which the MFT is greater than 15°C.

16. A composition as claimed in Claim 15 and of which the MFT lies within the range 20°C to 80°C.

17. A composition as claimed in any preceding claim in which methacrylic acid is the principal acidic monomer contributing to the emulsion polymer.

18. A composition as claimed in Claim 17 in which methacrylic acid represents at least 6% of the monomeric mixture from which the emulsion polymer is prepared.

19. A composition as claimed in Claim 18 in which styrene is copolymerised with a mixture of lower $\alpha\beta$-unsaturated carboxylic acids to form the emulsion polymer.

20. A composition as claimed in any preceding claim containing an emulsion polymer and up to 20% of emulsifiable polyethylene wax.

21. A composition as claimed in any preceding claim including ammonium carbonate or the carbonate/carbamate of morpholine, ethylene diamine, diisopropylamine, 3-methoxypropylamine or 2-amino-2-methyl-propan-1-ol.

22. A composition as claimed in any preceding claim containing up to 80% w/w of emulsion polymer, up to 20% w/w emulsifiable wax, 0—8% w/w plasticiser, up to 0.05% w/w levelling aid and of which the MFT is 15°C to 28°C.

23. A composition as claimed in claim 1 and substantially as described with reference to Examples 4 to 8 inclusive and Example 10.

24. A method for making a composition as claimed in claim 1 including the step of dehydrating a composition containing emulsion polymer, water-dispersible wax and emulsifier at a temperature less than the MFT characteristic of the composition.

25. A method for preparing a solid dehydrated coating compositions comprising the step of dehydrating a composition containing an emulsion polymer,' an emulsified wax and emulsifier, the dehydration step occurring above atmosphere temperature but below the MFT of the composition.

26. A method as claimed in claim 24 and 25 wherein the composition containing the emulsion polymer is dehydrated by air-drying on shallow trays.

27. A method as claimed in claim 24 or 25 wherein the composition containing the emulsion polymer is dehydrated by spray drying.

28. A composition as claimed in any one of claims 1 to 23 inclusive when

prepared by the method of claims 24, 25, 26 or 27.

29. A method as claimed in claim 24 and substantially as described with reference to the specific examples.

30. A combination comprising a composition as set forth in any one of claims 1 to 23 or claim 28 and a particulate solid material.

31. The combination set forth in claim 30 in which the particulate solid material comprises one or more detergent salts.

32. The composition set forth in claims 30 or 31 in which said particulate solid material comprises an alkaline material.

33. The combination set forth in claim 32 in which said particulate solid material comprises carbonate, bicarbonate, phosphate or silicate salts of ammonium, sodium or potassium.

34. A method for making the combination set forth in claims 30, 31, 32 or 33 comprising the steps of spraying drying the composition whilst simultaneously coating the particulate solid material therewith.

35. A method as claimed in claim 34 in which the particulate solid material is supported by a fluidized bed whilst the composition is spray coated thereon.

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